

NOVELTY - The hologram comprises an optically polymerizable chemical compound, an optical initiator and a thermosetting epoxy oligomer which is bonded to a pigment sensitizer. DETAILED DESCRIPTION - The hologram comprises an optically polymerizable chemical compound, an optical initiator

## BASIC-ABSTRACT:

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 DATE  
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 PUB-NO  
 APPL-DATE  
 JP 1161140A  
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 1997JP-325032  
 November 26, 1997

PATENT-FAMILY:  
 PUB-NO  
 JP 1161140 A  
 PUB-DATE  
 June 18, 1999  
 LANGUAGE  
 JA

PRIORITY-DATA: 1997JP-325032 (November 26, 1997)

PATENT-ASSIGNEE: TOPPAN PRINTING CO LTD[TOPP]

INVENTOR: ITO H; KUME M ; OE Y

TITLE:  
 Hologram recording material for gifts and security applications, etc, consisting of an optically polymerizable chemical compound, an optical initiator, a sensitizer and a pigment

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and a  
soluble thermosetting epoxy oligomer which is ionically bonded to a  
sensitizer. The oligomer is capable of cationic polymerization and  
comprises  
at least glycidyl and a polar group. The optical compound has  
olefinic  
group(s) which is liquid at normal temperature and has a boiling  
point more  
than 100°C at normal pressure and is capable of radical  
polymerization. The  
optical initiator generates Bronsted or Lewis acid, and activates  
cationic,  
radical polymerization when exposed to radiation. The pigment  
sensitizer makes  
the light initiator sensitive to the visible region.  
USE - Used as cover for magazine, gifts, credit cards, forgery  
prevention  
paper, etc.

ADVANTAGE - The hologram material has high sensitivity, chemical  
stability,  
good weather resistance, resolving degree, diffraction efficiency,  
transparency  
and long shelf life.

DESCRIPTION OF DRAWING(S) - The figure is an explanatory drawing  
showing the  
composition of the hologram recording material. (1) Hologram  
recording  
material; (2) Substrate; (3) Sensitization layer; (4) Protective  
layer.

CHOSEN-DRAWING: Dwg.1/2

TITLE-TERMS: HOLOGRAM RECORD MATERIAL GIFT SECURE APPLY CONSIST  
OPTICAL  
PIGMENT  
CHEMICAL COMPOUND INITIATE THERMOSETTING EPOXY OLIGOMER

DERWENT-CLASS: A89 G06 P84 V07

CPI-CODES: A05-A01E; A12-L02C; A12-L02E; G06-D; G06-E; G06-F03B;  
G06-F03D;

EPI-CODES: V07-F02C;

ENHANCED-POLYMER-INDEXING:  
Polymer Index [1.1]

D18 ; D01 D11 D10 D23 D22 D73 D42 F47; H0328; P0464\*R D01 D22  
 D42 F47;  
 H0237\*R; K9734; M9999 M2391; M9999 M2813;  
 Polymer Index [1.2]  
 018 ; G1570\*R G1558 D01 D11 D10 D23 D22 D31 D42 D50 D69 D73 D83  
 F47 7A;  
 G1161 G1150 G1149 G1092 D01 D11 D10 D19 D18 D32 D50 D76 D93 F32  
 F30  
 R00470 9523; P1898\*R P0464 D01 D11 D18 D19 D22 D42 D76 F34  
 F47;  
 H0011\*R; H0328; H0237\*R; M9999 M2391; M9999 M2813;  
 Polymer Index [1.3]  
 018 ; ND01; Q9999 Q8640 Q8606; K9778 K9745; Q9999 Q9029;  
 B4568; B9999 B4397 B4240; K9847\*R K9790;  
 B9999  
 B5332 B3372; K9676\*R; K9574 K9483; B9999 B5447 B5414 B5403  
 B5276;  
 Polymer Index [1.4]  
 018 ; S\* 6A; H0157;  
 Polymer Index [1.5]  
 018 ; D01 D12 D10 D11 D19 D18 D20 D23 D22 D75 D76 D78 D55 D51 D56  
 D57 D59  
 D61\*R D95 F08 F07 F17 F00 I\* 7A; H0226;  
 Polymer Index [1.6]  
 018 ; D01 D11 D10 D50 D83 F70 R00278 23; G1401 G1398 G4024 D01  
 D23 D22  
 D31 D42 D50 D65 D75 D84 F39 E00 E11 R00842 540; D01 D23 D22 D31  
 D41 D50  
 D76 D85 N\* 5A R00916 10; A999 A475; A999 A771;  
 Polymer Index [2.1]  
 018 ; G0022\*R D01 D51 D53 G0817\*R D54; H0000; H0011\*R; L9999  
 L2573  
 L2506; L9999 L2528 L2506; K9847\*R K9790;  
 Polymer Index [2.2]  
 018 ; G0908 G0873 G0817 D01 D51 D54 D57 D63 D11 D10 D26 D58 D92  
 F90 F41;  
 H0000; L9999 L2573 L2506; K9847\*R K9790;  
 Polymer Index [2.3]  
 018 ; ND01; Q9999 Q8640 Q8606; K9778 K9745; Q9999 Q9029;  
 B9999 B4580  
 B4568; B9999 B4728 B4568; B9999 B4397 B4240; K9847\*R K9790;  
 B9999  
 B5332 B3372; K9676\*R; K9574 K9483; B9999 B5447 B5414 B5403  
 B5276;  
 Polymer Index [2.4]  
 018 ; D01 D18\*R D61\*R; Ee 8B Tr; D01 F19 D23 D22 D76 D45; D01  
 D18\*R  
 D19 D18 D32 D76 D50 D61\*R D92 P\* 5A F\* 7A I\*; C999 C088\*R C000; C999

C293;  
 Polymer Index [2.5]  
 018 ; D01 D11 D10 D50 D83 F70 R00278 23; G1401 G1398 G4024 D01  
 D23 D22  
 D31 D42 D50 D65 D75 D84 F39 E00 E11 R00842 540; D01 D23 D22 D31  
 D41 D50  
 D76 D85 N\* 5A R00916 10; A999 A475; A999 A771;  
 Polymer Index [3.1]  
 018 ; P1707 P1694 D01;  
 Polymer Index [3.2]  
 018 ; ND01; K9676\*R; K9574 K9483; Q9999 Q7114\*R; K9712 K9676;  
 Q9999  
 Q8640 Q8606;

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			G 0 3 H 1/02	G 0 3 F 7/027	5 1 5

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(21) 出願番号	特願平9-325032	平成9年(1997)11月26日	(71) 出願人 000003193 凸版印刷株式会社 東京都台東区台東1丁目5番1号	(72) 発明者 伊藤 裕光 東京都台東区台東1丁目5番1号	(72) 発明者 久米 誠 株式会社内 東京都台東区台東1丁目5番1号 凸版印刷株式会社内	(72) 発明者 大江 尚 株式会社内 東京都台東区台東1丁目5番1号 凸版印刷株式会社内
(54) 【發明の名称】	ホログラム記録材料					

(57) 【要約】

【課題】光開始剤の増感剤に用いられるイオン性染料がハインダーやモノマーが溶解する一般的な有機溶剤には

溶解しにくく増感剤を適量混合できないためホログラム

記録材料の感度の低下、また混合しても相溶性がないた

めに凝集、析出する問題を解決し、化学的安定性、例

えば、耐光性、耐熱性、保存安定性に優れ、且つ感光性

が高くしかも感光速度の大きなホログラム記録材料を提

供する。

【解決手段】熱硬化性エポキシオリフィマー(A)と、光

重合性化合物(B)と、光開始剤(C)と、色増感剤

(D)とからなるホログラム記録材料は、可視光領域に

て増感可能なイオン性色増感剤(D)が、熱硬化性エ

ポキシオリフィマーの構造中にイオン結合していること

により、増感剤効率の向上、増感剤の揮発防止、ホログラ

ム記録材料の高感度化および高解像度化や化学的安定化

へと優れた特性を示す。



[illegible]

[illegible]





[illegible]





\*ルキノラム ヨーシト (Dイ3)、またはスチリル系色素「2-[[3-アシル-5-[[2-(1-エチル-4-((1H)-キノリニチン)エチル]2-アキニ-2-アチル]2-ニリチン]メチル]-3-エチル-4,5-ジフェニル]アラム フロロ」(Dイ4、5-ジフェニル]アラム フロロ)と、同様に着色色素を担持したエポキシアクリアートを調製し、実施例1-5と同様にホログラムを作製して、同所効率を測定した。その評価結果を表2に示す。

【表2】

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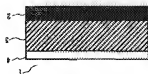
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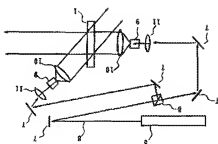
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【図1】



【図2】

1 ホログラム記録用媒体

【符号の説明】

【図2】 ホログラム撮影用の二光束光学系を説明する概

と記録用媒体の構成を説明する概略図である。

【図1】 本発明のホログラム記録材料からなるホログラ

【図面の簡単な説明】

可能なホログラム記録用媒体を提供できる。

明性などのホログラム特性値が良好なホログラムが作製

機械性及び保存安定性に優れ、かつ解像度、回折効率、透

10 11

10 レンズ

9

8 レンズ

7 ミラー

6 レンズ

5 レンズ

4 保護層

3 感光層

2 基板

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Notes:  
1. Untranslatable words are replaced with asterisks (\*\*\*\*).  
2. Texts in the figures are not translated and shown as is.

Translated 23:50:48 JST 10/21/2011  
Dictionary: Last updated 09/09/2011 / Priority: 1. Chemistry / 2. Medical/Pharmaceutical sciences / 3. Electronic engineering

## CLAIM + DETAILED DESCRIPTION

[Claim(s)]

[Claim 1] (A) Thermosetting epoxy oligomer which has respectively a glycidyl group and at least one or more ionicity dissociable groups in unit structures by solvent solubility and in which cationic polymerization is possible.

(B) Ordinary temperature, a photopolymerization nature compound which has at least one or more ethylenic unsaturated bonds whose boiling points it is a fluid in ordinary pressure, and are not less than 100 °C in ordinary pressure, and in which a radical polymerization is possible.

(C) A photoinitiator which generates Broensted acid or Lewis acid which activates radical species which will activate a radical polymerization if chemical action radiation is exposed, and cationic polymerization.

(D) It is an ionicity dye sensitizing agent in which sensitization is possible in a light range about a photoinitiator.

It is the hologram recording material provided with the above, and said ionicity dye sensitizing agent (D) is supported with an ionic bond in a structure of thermosetting epoxy oligomer.

[Claim 2] The hologram recording material according to claim 1 in which said thermosetting epoxy oligomer (A) is 400 or more weight per epoxy equivalents, and is characterized by being a solid in ordinary temperature ordinary pressure.

[Claim 3] The hologram recording material according to claim 1 or 2, wherein said thermosetting epoxy oligomer (A) is non-aromatic thermosetting epoxy oligomer.

[Claim 4] The hologram recording material according to claim 1 or 2, wherein said thermosetting epoxy oligomer (A) is bisphenol A type epoxy oligomer.

[Claim 5] The hologram recording material according to any one of claims 1 to 4, wherein said photoinitiator (C) is aromatic onium salt, an iron arene complex, or a triazine compound.

[Claim 6] The hologram recording material according to any one of claims 1 to 4, wherein said photoinitiator (C) is diaryliodonium salt.

[Claim 7] The hologram recording material according to any one of claims 1 to 6, wherein said ionicity dye sensitizing agent (D) is cyanine dye.

[Claim 8] The hologram recording material according to any one of claims 1 to 6, wherein said ionicity dye sensitizing agent (D) is a squarylium system pigment.

[Claim 9] The hologram recording material according to any one of claims 1 to 6, wherein said ionicity dye sensitizing agent (D) is a xanthene dye (thio).

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention starts the hologram recording material used for volume phase

type hologram formation, and it is high sensitivity to visible light especially argon laser radiation, or electron rays. It is related with a hologram recording material to excel in weatherability and preservation stability furthermore, and for hologram characteristics values, such as resolution, diffraction efficiency, and transparency, provide a good hologram.

[Description of the Prior Art]Conventionally, since regeneration of three-dimensional stereoscopic films is possible for a hologram, it is used for covers, such as books and a magazine, the POP display, the gift, etc. from the outstanding design nature and the ornament effect. Since it can say that a hologram is equivalent to the information on a submicron unit, it is used for the mark for forgery prevention, such as negotiable securities and a credit card, etc. Since especially the volume phase type hologram can modulate a phase, without absorbing the light beam which passes an image by forming the spatial interference fringes from which the refractive index instead of optical absorption difference differs into a hologram medium. In recent years, the application to the hologram optical element (HOE) represented by the head up display (HUD) for automobile loading other than a display use is expected.

[0003]By the way, a volume phase type hologram recording material is exposed by high sensitivity to a laser beam with a visible oscillation wavelength, and it is required that high definition should moreover be shown. It is required that actually produced characteristics, such as diffraction efficiency of a hologram, the wavelength reproducibility of regeneration light, and a band width (regeneration light full width at half maximum), should suit the purpose of use. To the hologram recording material for HOE, diffraction efficiency with 5000-6000 spatial frequency/mm especially desirable for the full width at half maximum (band and width) of regeneration light to be less than 5 nm from photography wavelength, and to excel in preservation stability over a long period of time is also needed further.

[0004]The general principle about hologram production is written in some document and the technical books of Chapter 2, for example, a "holographic display" (the volume for Junpei Tsujuchi; Sangyo Publishing). According to these, it is put on the coherent position of two luminous flux which irradiates a recording object thing with one side of a laser beam, and can generally receive the total reflection light from it, photosensitive recording medium, for example, dry plate for photographs. Another coherent light other than the reflected light from a subject is directly irradiated by the recording medium, without hitting a subject. Object light and the light directly irradiated with the reflected light from a subject by the medium are called reference beam, and the interference fringe of a reference beam and object light is recorded as picture information. Next, if the produced hologram is observed under suitable Lighting Sub-Division, the object image which the light from an illumination light source was diffracted by the hologram so that the wave face of the reflected light which reached the recording medium first from the subject on the occasion of record might be reproduced, and resembled the real image of the subject as a result will be observed in three dimensions. The hologram which enters a reference beam and object light in a recording medium from the same direction, and is formed is known as a transmission type hologram. Generally the hologram which was entered and was mutually formed from the opposite side of a recording medium on the other hand is known as a reflection type hologram. A transmission type hologram can be obtained by a publicly known method which is indicated, for example in the US,3506327 B

gazette, the US,3642787 B gazette, etc. A reflection type hologram is producible by the publicly known method indicated by the US,3524406 B gazette, for example. [0005]There are refractive-index abnormal conditions as a value which compares the hologram formed as an image. This is a value of the incident light diffracted by the diffraction grating specified from diffraction efficiency and the thickness of a recording medium namely [ comparatively ], when it irradiates with two luminous flux so that a medium and the angle to make may become the same at a recording medium, and a diffraction grating is produced. Refractive-index abnormal conditions



are the quantitative measures of change of the refractive index produced in the exposure part of a volume type hologram and an unexposed part, i.e., the portion which light interferes and suits in slight strength, and the portion weakened mutually.

It can ask by the theoretical formula [Bell,Svt.Tech.,J., 48 and 2909, (1969)] of a KOGERNICK (H. Kogelnik).

Compared with a transmission type hologram, it is high-resolution, namely, generally, since a reflective phase type hologram has many interference fringes formed in per mm, it is difficult to record, and it is difficult to obtain high refractive-index abnormal conditions.

[0006]Generally as a recording material of such a volume phase type hologram, the sensitive conventionally, The sensitive material of a dichroic acid gelatin system is a material most widely used for recording volume phase type hologram with the high diffraction efficiency and low noise characteristics. However, this sensitive material has a short shelf life, and must be prepared to the degrees which is production, in order to perform wet developing, in swelling and the contraction process of gelatin which it is needed in the case of hologram production, it is accompanied by modification of a hologram. For this reason, it also has the problem that reproducibility is bad. A silver salt sensitized material is not a sensitive material which needs complicated treatment and this can also satisfy from a viewpoint of stability and workability after record. Each of these sensitive materials had the problem of being inferior to an environmental capability—proof, for example.

[0007]On the other hand, the hologram recording material using poly-N-vinylcarbazole as a material provided with the characteristics that it should excel in an environmental capability—proof, and should have hologram recording materials, such as high resolution and high diffraction efficiency, is raised. For example, the hologram recording material which consists of an annular \*\*\*\*-alpha-dicarboxyl compound and a sensitizing agent (JP.S60-45283A). The hologram recording material which consists of 1,4,5,6,7,7-hexachloro-5-norbornene anhydrous-2,3-dicarboxylic acid and a pigment (JP.S60-227280A). The hologram recording material (JP.S60-260080A) which consists of 2,3-norbornane dione and thioflavin, the hologram (JP.S62-123489A) which consists of thioflavin T and iodoforn, etc. are proposed. Since these hologram recording materials need wet developing too, a complicated treatment process is needed and it has the problem of being inferior to reproducibility. Since it is the sensitive material which made poly-N-vinylcarbazole bases resin, it is chemical stability, and although it excelled in high resolution and an environmental capability—proof, it crystallizes, and is very easy to whiten poly-N-vinylcarbazole, and the reproducibility of transparency has the problem that it will be bad and a solvent will also be resisted. Still much more improvement is desired in the sensitivity characteristic.

[0008]The optical hardening resin constituent using the photoinitiator which composes the combination of 3-oxo-coumarin and diaryliodonium salt as a material can carry out optical hardening by high sensitivity (JP.S60-88005A). The hologram recording material (JP.H4-31590A) which combining polymethylmethacrylate as this photopolymerization initiator and a support polymer in the peak wavelength of regeneration wavelength, or the half band width of peak wavelength, and in the case of development in order to make an opening form by a wet process although it is chemically stable and has high resolution and high sensitivity, it had the problem that development nonuniformly occurred easily. Since many openings existed in the hologram, it had the problem of being inferior to a heat-resistant property and heat-resistant pressure nature.

[0009]The photopolymerization type hologram recording material which can produce a hologram is indicated in the US.3939485B gazette and the US.3685826B gazette by 1 time of a treatment process without a wet process to this problem. The former has a sensitive material of two types and, [as the 1st example] reactivity and a refractive index — things — the combination of the unsaturated ethylenic monomer and photopolymerization initiator in which two polymerizations are

It is shown and he is eternal volume phase type Jolo Crad by 1-time exposure of chemical action matrix.

monomer and photopolymerization initiator in which photopolymerization is possible into the polymer becomes from the hologram recording material in which the No. 6 gazette blended the ethylenic 1001110011100111. Manufacture \*\*\*\*\* of the stable hologram which latter United States patent 3658th which example and viscosity is low, and reproducibility — — — many — the problem remains.

heard to punch to a substrate, and a thick film, since it is a mixture of low molecular weight also in completion does not have preservation stability. workability, such as being hard to form that it is Phenylphthalate exists in a system as a compound of low molecular weight, and after hologram abnormal conditions are not obtained. 1 which is a nonresponsive compound in the 2nd example — polymerization takes place, and, as for what was shown in the 1st example, high refractive-index — there are the following problems. Namely, a certain amount of [ a reactant low monomer ] recorded with the difference of a refractive index, and a volume phase type hologram is formed with strong light intensity again at a portion with weak light intensity. Thus, an interference fringe is a reactant light monomer diffuses a reactant low monomer or nonresponsive compound into a portion to two to luminous flux becomes strong, and the concentration gradient of a monomer arises and a progresses more in the portion to which the light intensity of the interference fringe made according if it uses with which photoreactive resin composition, the polymerization of a reactant high monomer phenylphthalate and benzoin methyl ether, and can produce a hologram like the 1st example. Even ethylene glycol dimethacrylate, 1 — 1 is a photoreactive resin composition which consists of which differs in a refractive index, and a polymerization initiator. For example, butyl methacrylate, agree when it polymerizes, and four ingredients of two monomers, the nonresponsive compound which can be polymerized and the unsaturated ethylenic monomer which works as a cross linking unsaturated ethylenic monomer which has a comparable refractive index as the 2nd example and the this in the glass plate of two sheets, and exposing by a luminous-flux optical system. The can carry out hologram record by consisting of vinylcarbazole and benzoin methyl ether, sandwiching possible. For example, cyclohexyl methacrylate, N — 1 is a photoreactive resin composition which

The complete exposure of the continuing chemical action during recording is fixed to the hologram formed, although the hologram recording material indicated here is \*\* to give many advantages in respect of workability, reproducibility, etc., the diffraction efficiency is low, in this hologram recording material, the range of the refractive-index abnormal conditions of the completed hologram is 0.001 to 0.003. As a result, the reconstruction image of the formed hologram has only the limited luminance. Although it is also possible to give a certain amount of luminance by thickening a hologram recording layer, this solution brings a result for which a manufacturer is made to use a lot of hologram recording materials, and when making it fix in glass laminates, such as a certain medium, for example, the head up display for mount, etc., and using, it causes trouble. The hologram formed of this should be taken notice of that decline in diffraction efficiency generally takes place by prolonged preservation.

[0012]As an improvement art also including the manufacturing method of the hologram recording material indicated by this US,3658526,B gazette, the US,5098803,B gazette, JP,H2-3081,A, and JP,H2-3082,A are indicated. Thermoplastics, the unsaturated ethylenic monomer which can be polymerized, and a photopolymerization initiator are made into basic composition, and in order to raise refractive index, abnormal conditions, the work which gives refractive index difference to either the thermoplastics or the unsaturated ethylenic monomer which can be polymerized using the compound which has an aromatic ring is carried out. However, like what is indicated in the US,3658526,B gazette, since resin of the amount of polymers is used as binder MATOKUSSU, the diffusibility of the monomer at the time of exposure is restricted, many light exposures are needed and high diffraction efficiency cannot be acquired. In order to improve this

point, the nonresponsive plasticizer is added, but it has a problem about the film hardness of the formed hologram by this use, and the plasticizer which is nonresponsiveness exists in a system as a compound of low molecular weight, and after hologram completion does not have preservation stability. In addition, since the carrier holding this is thermoplastics, it has a fault inferior to a heat-resisting property.

[0013] On the other hand, according to JP,H5-107999,A which is the improvement art of the above-mentioned fault, instead of the plasticizer in the above-mentioned patent, cation pile affinity monomer and a cationic initiator are blended, and the problem by a nonresponsive plasticizer remaining after hologram formation is solved. However, since light irradiation remarkable for fixing spread in the case of fixing, distortion arises in the formed hologram and high diffraction efficiency cannot be acquired. As well as conventional technology since the carrier holding this is thermoplastics, it has a fault inferior to a heat-resisting property, workability, such as being hard to form that it is hard to punch to a substrate, and a thick film as a carrier for holding, by the system which does not use thermoplastics, since viscosity is low, and reproducibility is low — many — the problem remains.

[0014] The photosensitive resin composition for hologram record which consists of an epoxy resin, a radical polymerization nature unsaturated ethylenic monomer, and an optical radical polymerization agent is indicated by JP,H5-94014,A. As long as an working example is seen, two kinds of epoxy resins are used, but in order to require the complicated work of performing a radical polymerization and cationic polymerization with the light of a separate wavelength band when an ultraviolet curing nature epoxy resin is used, and to prepare the diffusibility of a monomer, fine tuning of raising viscosity according to pre-exposure is needed, and workability and reproducibility are difficult. Workability is very bad in order for hardening of the epoxy resin for fixing to take remarkable ultraviolet curing and heat time, when thermoset epoxy resin and a curing agent are used. In addition, high diffraction efficiency cannot be acquired in the improvement art indicated here. In this way, the necessity over the photopolymerization type hologram recording material in which it was improved for hologram record, and the optical element using it exists.

[0015] In consideration of this point, the common No. 261640 [ seven to ] gazette is indicated as a hologram recording material improved further. According to the above-mentioned patent, it consists of combination of solid epoxy oligomer and an ethylenic monomer by ordinary temperature ordinary pressure, and the hologram recording material excellent in the resistance to environment, especially the heat-resisting property is indicated.

[0016] By the way, although the photopolymer various type is put in practical use now, the photopolymer of a cinemate system is photorealist which is developed most for many years and used for production of a printed circuit etc. still now. On the other hand, optical hardening type printing ink, a solder resist, etc. are various, and oligomer and polymer of acrylic are used. As for these, in any case, the sensitivity in non-sensitization status is low, and, respectively, As an effective sensitizer of a cinmanon acid ester system, 5-nitrocenaphthene, N-acetyl-4-nitro-1-naphthylamine, PIKURAMIDO, etc. are added, and benzoin, ketals, and anthraquinone are added as a photopolymerization initiator of acrylic. However, in the application process of a photopolymer, and a prebaking stage (after postbake process), a low-molecular sensitizer and a stabilizer may coze out on the polymer film surface, and may sublimate during the air further. In the solder resist and other surface coating materials after paint film hardening, a photopolymerization initiator unreleased during the use may deposit on the surface, such a phenomenon not only reducing the sensitivity of a photopolymer, and the reproducibility of workability, but imitating aggregation of work environment, and the fall of the reliability of parts — Lycium chinense — it becomes.

[0017] In recent years, polymerization of a sensitizer has been studied as one of the methods of solving these problems. In the photochemical reaction of an organic compound, [ polymerization of a sensitizer ] in [ from the field of improvement in recovery, reuse, and sensitizer efficiency of

isolation of the product from the system of reaction, the simplicity of refining, and a sensitizer] a photopolymer, in the application to the polymeric material [ viewpoint / of the prevention from volatilization of a sensitizer, a raise in the sensitivity of polymer, and high-resolution of further others, polymerizing and its practical use of the sensitizer attract attention from the Reasons of improvement in a material property, stabilization of polymer, etc. in research of the latest polymer sensitizer, it does not stop at polymerization of a simple sensitizer, but even if it compares with a low-molecular sensitizer by controlling the introductory rate or designing a reaction field, the polymer sensitizer and the system of reaction which show the outstanding sensitization effect are beginning to be found out.

[0018]

[Problem to be solved by the invention] However the hologram recording material conventionally included including the above-mentioned hologram recording material [ It is necessary to dissolve parts for a solid presentation, such as a binder and an initiator system, and to make it uniform by parts for a fluid presentation, such as a monomer, at the time of recording-medium production for obtaining a suitable hologram, without dissolving a binder, a monomer, and an initiator system uniformly with a suitable solvent, or using a solvent. A binder and a monomer dissolve in neither water nor alcohol as a solvent, although it dissolves in a common organic solvent. On the other hand, [ ionic dye including a xanthene pigment useful (this) as a sensitizer of a photoinitiator ] Since it is most which is solved a little to the common organic solvent which the binder and monomer of what is dissolved dissolve in adequate amount mixing of the sensitizer cannot be carried out, it has the problem that the sensitivity of the sensitized material obtained falls and it mixes them, there is a problem of condensing and depositing.

[0019] It is in this invention having perceived such a problem, and having been made, and the place made into the problem being excellent in chemical stability, for example, lightfastness, a heat-resisting property, and preservation stability, and photosensitivity providing a hologram recording material with exposure speed big high moreover.

[0020]

[Means for solving problem] This invention persons result in this invention, as a result of repeating research wholeheartedly, in order to solve the above-mentioned problem.

[0021] Namely, the thermosetting epoxy oligomer to which the invention according to claim 1 has respectively a glycidyl group and at least one or more ionic dissociable groups in unit structures by (A) solvent solubility and in which cationic polymerization is possible. By ordinary temperature and ordinary pressure, with a fluid (B) And the photopolymerization nature compound which has at least one or more ethylenic unsaturated bonds whose boiling points are not less than 100 °C in ordinary pressure, and in which a radical polymerization is possible, (C) The photoinitiator which will generate the Brønsted acid or Lewis acid which activates the radical species which activate a radical polymerization, and cationic polymerization if chemical action radiation is exposed, (D) In the hologram recording material which consists a photoinitiator of an ionic dye sensitizing agent in which sensitization is possible in a light range, a dye sensitizing agent (D) is the hologram recording material currently supporting with the ionic bond in the structure of thermosetting epoxy oligomer, [0022] The hologram recording material according to claim 2 is characterized by thermosetting epoxy oligomers (A's) being 400 or more weight per epoxy equivalents, and being a solid in ordinary temperature ordinary pressure based on the invention of Claim 1.

[0023] The hologram recording material according to claim 3 is characterized by thermosetting epoxy oligomer (A) being non-aromatic thermosetting epoxy oligomer based on an invention of Claim 1 or

either of 2.

[0024] The hologram recording material according to claim 4 is characterized by thermosetting epoxy oligomer (A) being bisphenol A type epoxy oligomer based on an invention of Claim 1 or either of 2.

[0025] The hologram recording material according to claim 5 is characterized by a photoinitiator (C)

being aromatic onium salt, an iron arene complex, or a triazine compound based on one invention of the Claims 1-4.

[0026] The hologram recording material according to claim 6 is characterized by a photoinitiator (C) being diaryliodonium salt based on one invention of the Claims 1-4.

[0027] The hologram recording material according to claim 7 is characterized by an ionicity dye sensitizing agent (D) being cyanine dye based on one invention of the Claims 1-6.

[0028] The hologram recording material according to claim 8 is characterized by an ionicity dye sensitizing agent (D) being a squarilium system pigment based on one invention of the Claims 1-6.

[0029] The hologram recording material according to claim 9 is characterized by an ionicity dye sensitizing agent (D) being a xanthene dye (thio) based on one invention of the Claims 1-6.

[0030]

[Mode for carrying out the invention] Hereafter, this invention is explained in detail. Drawing 1 is a schematic diagram explaining the composition of the medium 1 for hologram record which consists of a hologram recording material of this invention, and drawing 2 is an approximate account figure explaining the 2 luminous-flux optical system for hologram photography.

[0031] The thermosetting epoxy oligomer which has respectively a glycidyl group and at least one or more ionicity dissociable groups in unit structures by the component (A) solvent solubility used by this invention and in which cationic polymerization is possible can be obtained by introducing an ionicity dissociable group into usual thermosetting epoxy oligomer 1 as this usual thermosetting epoxy oligomer. For example, bisphenol A and bisphenol A D, bisphenol B, Bisphenol A F, the bisphenol S, bromo-ized bisphenol A, it is manufactured by the condensation reaction of various bisphenol compounds, such as \*\*\*\* bisphenol A and \*\*\*\* bisphenol A D, the \*\*\*\* bisphenol B, the \*\*\*\* bisphenol A F, and formation of a \*\*\*\* bromo, and epichlorohydrin, it is considered as an example and a commercial item is mentioned, Araldite6071, 6084, 6097 and 6099 (CIBA), Dow661, 664, 667 (Dow). [Epicat 1001, 1002, 1004, 1007, 1009, 1010 and 1100L (oil) recovery shell company]. [Although there are Chemical, YDB 500, 406, 408, and 412 and ZX1417, 1413, ST3150, 5080 (Tohto Kasei Co., Ltd.), EBPS-300 (Nippon Kayaku Co., Ltd.), etc., it is not limited to these, it can also use in two or more sorts of combination, such as this.

[0032] As an ionicity dissociable group, although a carboxyl group, a sulfonic group, an amino group, a phosphonium salt residue, a sulfonium salt residue, various metal ion content groups, etc. can be mentioned, it is not limited to these. The method of introducing these ionicity dissociable groups into the above-mentioned epoxy oligomer can be acquired by the reaction of the compound and epoxy oligomer which have an ionicity dissociable group.

[0033] As a photopolymerization nature compound which is used by this invention and in which a component (B) radical polymerization is possible, at least one or more unsaturated bonds of ethylene nature may be included in a structural unit, a polyfunctional vinyl monomer other than the vinyl monomer which is one organic functions may be included, and they may be these mixtures. Specifically Acrylic acid (meta), itaconic acid, maleic acid, acrylamide (meta), [ high boiling point vinyl aliphatic series polyhydroxy compound, for example, ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, propylene glycol, dipropylene glycol, tripropylene glycol, a tetrapropylene glycol, Neopentyl glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,10-Decan diol, or poly(methyl acrylate) ester, such as trimethylolpropane, pentaerythritol, dipentaerythritol, sorbitol, and mannitol. Or as for mono-\*\*\*\*, such as an allycyclic polyhydroxy compound, for example, JISHIKURO pentanol, JISHIKURO pentanol, and tricyclodecanedimethyloxy, (meta) acrylate, although polyethylene glycol (meta) acrylate or polypropylene glycol (meta) acrylate can be mentioned, it is not limited to these.

[0034] As a photoinitiator which generates the Broensted acid or Lewis acid which activates the radical species which will activate a radical polymerization if the component (C) chemical action radiation of this invention is exposed, and cationic polymerization] J. Photopol. Sci. Technol., 2, the

salt is especially preferred at ROGENO methylation-s-triazine, iodonium salt, as an example of the diazoniomium salt; a seleno NIUMJ salt s-resonance iodonium salt, etc. can be mentioned. [as an example of the dihydroiodonium salt used by this invention] Macromolecules, 10, a compound given in 1307(1977),  
For example, diphenyl(iodonium, ditcyl iodonium, phenyl p-arsyl) iodonium, Bis-(m-nitrophenyl)  
iodonium, bis(p-tert-butylphenyl)(iodonium), Alkylchloride of iodonium, such as bip -

chlorophenyl(iodonium bromide or Howe fluoride salt, a hexafluorophosphate salt,  
hexachloroarsenate salt, etc. can be mentioned, it is not limited to these.

[0035] The component (D) photoindicator of this invention can be mentioned for a xanthene dye  
(thio), cyanine dye, a squarylium system pigment, a styryl system pigment, loader cyanine dye, etc.  
as an ionic dissociation nature dye sensitizing agent in which sensitization is possible in a light

[0036] As an example of a xanthene dye (thio), first, the rhodamine 110, the rhodamine 123, rhodamine 6G, The rhodamine 116, rhodamine B, the rhodamine 19, fluorescein, Eosine, erythrosine, a basic red 1, the rosin 2G, the rhodamine 4G, the C.I. basic violet 10, the rhodamine 120F, the C.I. basic red 1, the rosine red 3B. Although the pyronin G, rhodamine SHARAHNAAG, the C.I. acid red 52, the rhodamine S, etc. can be mentioned, it is not limited to these.

[0037] As an example of cyanine dye [ NK-663 by a Japanese sensitizing dye laboratory company, NK-3989, NK-719, NK-6, NK-85, NK-1046, NK-1723, NK-1538, NK-2203, NK-1952, NK-1420, NK-2610, NK-76, NK-382, NK-1056, [ NK-616, NK-716, NK-5, NK-138, NK-741, NK-1836, NK-3988, NK-322, NK-2764, NK-3620, NK-3962, NK-3618, NK-1210, NK-2150, and NK-734 grade ] Although it can mention, it is not limited to these.

[0038] Although NK-3912 by a Japanese sensitizing dye laboratory company, NK-3892, NK-3905, NK-2848, NK-2990, and NK-3906 grade can be mentioned as an example of a squarylium system pigment, it is not limited to these.

[0039] As an example of a styryl system pigment, it is 2-[2-[(4-(dimethylamino) phenyl) ethynyl]-3-

[illegible][illegible]

[0042]Furthermore, additives, such as thermal-polymerization inhibitor, a chain transfer agent, and an antioxidant, can also be added to the hologram recording material of this invention if needed.

[0043]The sensitizing solution which each of these above-mentioned components suitably and was mixed and obtained at an arbitrary rate A spin coater, which carries out coat formation on the substrates 2, such as a glass plate, a polymethylmethacrylate board, and a polyester film, and produces the medium 1 for hologram record using publicly known coating means, such as a roll coater and burr KOT-A. At this time, the protective layer 4 may be formed as an oxygen obliteration film on the photosensitive layer 3. Films, glass, etc., such as what is equivalent to the above-mentioned substrate 2 or polyolefine, polyvinyl chloride, polyvinylidene chloride, polyvinyl alcohol, or polyethylene terephthalate, can be used for the protective layer 4, for example. When applying a sensitizing solution, it may dilute with a suitable solvent if needed, but desiccation is required after applying on a substrate in that case.

[0044]Drawing 2 is a schematic diagram explaining the 2 luminous-flux optical system for reflection type hologram photography, and the laser beam 6 oscillated from the laser 5 is irradiated by the medium 1 for hologram record which consists of a hologram recording material of this invention via the mirror 7, the beam splitter 8, the SUPERISHARU filter 9, and the lens 10. Although this invention does not carry out detailed explanation and illustration, it is possible similarly about production of a transmission type hologram, and the transmission type hologram which has hologram characteristics outstanding by this can be obtained.

[0045]As a light source suitable for the hologram recording material of this invention, although a helium cadmium laser, argon laser, a krypton laser, He Ne laser, etc. can be used, it is not limited to this.

[0046]Hereafter, a concrete working example explains this invention still in detail.

It dissolving in 1007 g of the <working-example 1> thermosetting epoxy oligomer (made by a trade name "Epiccoat 1007", oil recovery shell epoxy company), and 200 g of N, N dimethylformamide, and 7 g of succinic anhydride and pyridine were added, and it agitated until it became uniform. It heated at 60 more \*\* for 2 hours. It was made to precipitate after radiational cooling and in methanol, and the epoxy oligomer which has the target ionicity dissociable group (here capboxyl group) was obtained. 20g of epoxy oligomer (made by a trade name "NK-2125", Japan sensitizing dye laboratory company) which have this ionicity dissociable group were dissolved in DMF50ml, and it agitated at 40 \*\* for 3 hours. Methanol was filled with this solution after radiational cooling, and operation of obtaining the epoxy oligomer which flowed into methanol and colored it was performed several times, and was refined.

[0047]What carried out the mixture solution of this polymer 100 weight section, triethylene-glycol-diacylate 50 weight section, and the diphenyliodonium hexafluorophosphate 10 weight section to 2-100 weight section was used as the sensitizing solution. Applicator was used, it applied and dried to the glass substrate, and the photosensitive layer was formed so that dry membrane thickness might be set to about 15 micrometers in this sensitizing solution. Then, the photosensitive layer top was covered by the polyvinyl alcohol (PVA) film, and the medium for hologram record was produced.

[0048]After exposing the medium for hologram record according to the 2 luminous-flux optical system for hologram photography shown in drawing 2, using a krypton laser (647.1 nm) as a light source and forming a hologram picture, heat-treatment was performed at 100 \*\* for 30 minutes.

[0049]The diffraction efficiency of the obtained hologram was measured with the spectrograph multi-meter made from Jasco Industry. This spectrophotometer can install a photograph multi-meter with a 3-mm-wide slit on the circumference with a radius [ centering on a sample ] of 20 cm. Measurement conditions entered the 0.3-mm-wide monochromatic light into the sample at the angle of 45 degrees, and detected the diffraction light from a sample. The ratio of the biggest value and the time

of receiving direct incident light, without placing a sample was made into diffraction efficiency except specular reflection light. The evaluation result is shown in Table 1.

[0060] instead of the cyanine dye "NK-2125" Japan sensitizing dye laboratory company make which is an ionicity dye sensitizing agent of the <working-example 2-4> working example 1

Respectively Squarylium system pigment "NK-3905" Japan sensitizing dye laboratory company make, A styryl system pigment "2-[2-[4-(dimethylamino) phenyl]-1,3-butadienyl]-1-ethynaphth [1,2-] [d] this ZORUMU YOJITO" (Dye1), or — a loader — cyanine dye — "three — ethyl — two — [ — three — ethyl — five — [ — two — (1-ethyl-2(1H)-quinolinylidene) — ethylidene — ] — 4-oxo — two — thia — [ — two — thia — ZORUMINRIDEN — ] — methyl — ] — benzo — thia — ZORUMU — YOJITO" — (Dye2) — using. Except it, the pigment was introduced into epoxy oligomer like the working example 1, the hologram was produced, and diffraction efficiency was measured. The evaluation result is shown in Table 1.

[0061]<Comparative example> thermosetting epoxy oligomer (made by trade name "Epicoat 1007" triethylene-glycol-diisocylate 50 weight section and diphenyliodonium hexafluorophosphate 10 weight section, and the squarylium system pigment "NK-3905" Japan sensitizing dye laboratory company make 50 weight sections to 2-butnone 100 weight section was used as the sensitizing solution. Although this sensitizing solution was applied to the glass substrate using applicator so that thickness might be set to about 15 micrometers, and the photosensitive layer was formed, since the squarylium system pigment "NK-3905" hardly dissolved in 2-butnone when stoving is carried out, hologram production by a visible light laser was not completed.

[0062]

[Table 1]

実施例 1	NK-2150	5.0	8.2
実施例 2	NK-3905	5.0	8.5
実施例 3	Dye1	8.0	8.9
実施例 4	Dye2	8.0	8.1

[0053]It dissolved in 100 g of the <working-example 5> thermosetting epoxy oligomer (made by the trade name "Epicoat 1007" oil recovery shell epoxy company), and 200 g of N, N dimethylformamide, and 2-bromoethane sulfone sodium 10g and NaOH5g were melted and added to 50 ml of water. It cooled, after making this react at 100 °C at 5:00. This is poured out under water, and was settled and the epoxy oligomer which has the target ionicity dissociable group (here sulfonic group) was obtained. 20g of epoxy oligomer and 0.1 g of cyanine dye (made by a trade name "NK-529" Japan sensitizing dye laboratory company) which have this ionicity dissociable group were dissolved in DMF50ml, and it agitated at 40 °C for 3 hours. Methanol was filled with this solution after radiational cooling, and epoxy colored oligomer was obtained. This was dissolved in N, N dimethylformamide, and operation of obtaining the epoxy oligomer which flowed into methanol and colored it was performed several times, and was refined.

[0054]What carried out the mixture solution of this polymer 100 weight section, monomer (made by trade name "screw coat #192" OSAKA ORGANIC CHEMICAL INDUSTRY, LTD.) 50 weight section, and the diphenyliodonium hexafluorophosphate 10 weight section to 2-butnone 100 weight section was used as the sensitizing solution. Applicator was used, it applied and dried to the glass substrate, and the photosensitive layer was formed so that dry membrane thickness might be set to about 15 micrometers in this sensitizing solution. Then, the photograph record was covered by the polyvinyl alcohol (PVA) film, and the medium for hologram record was produced. Diffraction efficiency was measured like the working example 1. The evaluation result is shown in Table 2.



[0055] instead of the cyanine dye (made by a trade name "NK-529" Japan sensitizing dye laboratory company) of the <working-example 6-9> working-example 5] Respectively Cyanine dye (made by a trade name "NK-138" Japan sensitizing dye laboratory company, A squarylum system pigment -4-[4-(dimethylamino) phenyl]-1,3-butadienyl]-1-ethyl kino RIUMU YOUTO) (Dye3), or -- styryl -- a pigment -- two -- [ -- three -- allyl- -- five -- [ -- two -- (1-ethyl-4(H)-quinolinylidene) -- ethylidene -- ]-4-oxo -- two -- thia -- ZORJINIRIDEN --] -- methyl -- ]-3-ethyl- -- 4,5 -- diphenyl -- thia -- ZORJUMU -- ] (Dye4). Except using the epoxy oligomer which supported various pigments like the working-example 5 from the working-example 1 was prepared, the hologram was produced like the working-example 1-5, and diffraction efficiency was measured. The evaluation result is shown in Table 2.

[Table 2]

実施例 5	NK-529	5.0	8.6	実施例 6	NK-138	5.0	8.4	実施例 7	NK-3912	5.0	8.1	実施例 8	Dye3	5.0	8.7	実施例 9	Dye4	5.0	8.2
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[Table 3]

実施例 10	エリスロシンB	8.0	8.1	実施例 11	ローズマリン	8.0	8.4
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[0057] It dissolved in 100 g of the <working-example 10> thermosetting epoxy oligomer (made by trade name "Epicote ZX1413" Tohto Kasei Co., Ltd.), and 200 g of N, N dimethylformamide, and 10 g making this react at 100 °C for 5 hours. This is poured out under water, and was settled and the epoxy oligomer which has the target ionic dissociable group (here amino group) was obtained. 20 g of epoxy oligomer which has this ionic dissociable group, and (thio) xanthene dye "erythrosine B" 0.1 g were dissolved in DMF 50 ml, and it agitated at 40 °C for 3 hours. Methanol was filled with this solution after radiation cooling, and epoxy colored oligomer was obtained. This was dissolved in N, N dimethylformamide, and operation of obtaining the epoxy oligomer which flowed into methanol and colored it was performed several times, and was refined. [0058] What carried out the mixture solution of this polymer 100 weight section, monomer (made by trade name "HX220" Nippon Kayaku Co., Ltd.) 50 weight section, and the diphenyliodonium hexafluorophosphate 10 weight section to 2-butanol 100 weight section was used as the photosensitive layer was formed so that dry membrane thickness might be set to about 15 micrometers in this sensitizing solution. Then, the photosensitive layer top was covered by the polyvinyl alcohol (PVA) film, and the medium for hologram record was produced. Diffraction efficiency was measured like the working-example 1. The evaluation result is shown in Table 3. [0059] Except using a "rose bengal" instead of the xanthene dye "erythrosine B" which is an ionic dye sensitizing agent of the <working-example 11> working-example 10 (thio), the sensitizing solution was prepared like the working-example 10, and the hologram was produced. The result is shown in Table 3.

[0060]

[0061] Even if it neglected the hologram of the working example 1-1 under the environment of 10 hours at 180 days and 150 \*\* by 25 \*\* and 60%RH, the decline in diffraction efficiency was not accepted.

[0062]

[Effect of the invention](A) The thermosetting epoxy oligomer which has respectively a glycidyl group and at least one or more ionicity dissociable groups in unit structures by solvent solubility and fluid (B) And the aliphatic series monomer which has at least one or more ethylenic unsaturated bonds whose boiling points are not less than 100 \*\* in ordinary pressure, and in which a radical polymerization is possible, (C) The photoinitiator which generates the Broensted acid or Lewis acid which activates the radical species which will activate a radical polymerization if chemical action radiation is exposed, and cationic polymerization, (D) In the hologram recording material which consists a photoinitiator of an ionicity dye sensitizing agent in which sensitization is possible in a light range, [ an ionicity dye sensitizing agent (D) ] [ by being supported with the ionic bond in the structure of the thermosetting epoxy oligomer ] It is a hologram recording material in which outstanding characteristics, such as improvement in sensitizer efficiency, prevention from volatilization of a chemical stabilization, are shown, The medium for hologram record by which it excels in the weatherability and preservation stability using this, and hologram characteristics values, such as resolution, diffraction efficiency, and transparency, can produce a good hologram can be provided.

[Translation done.]